



PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of  
Kazuhiko TACHIBANA, et al.

Docket No: Q37866

Appln. No.: 09/500,446

Group Art Unit: 1711

Confirmation No.: 2630

Examiner: Melanie D. Bissett

Filed: February 05, 2000

For: WATERSTOP SEALING MATERIAL

DECLARATION UNDER 37 C.F.R. § 1.132

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

SIC:

I, Kazuhiko Tachibana, hereby declare and state:

THAT I am a citizen of Japan; and

THAT I am a co-inventor of the invention disclosed and claimed in the above-identified application.

I have performed or have had performed under my direct supervision and control the experimentation described below.

EXPERIMENTATION

Into a four-necked separable flask equipped with an agitator, a temperature indicator and a water-separating tube were charged 290 g of polyacrylonitrile which is liquid at room temperature (PLACCEL L212AL produced by Daicel Chemical Industries, Ltd.; hydroxyl number: 39.4 KOH mg/g) (hydroxyl group: 0.919 equivalent), 11.94 g of succinic anhydride

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(acid group: 0.319 equivalent), and as a catalyst 79 mg of dibutyltin oxide (abbreviated as DBTO hereinafter) (0.1 equivalent %). In the presence of a small amount of toluene as a solvent for discharging reaction water, the mixture was heated with stirring to a temperature of 180°C and then kept at the temperature. After a while, water was discharged and separated, showing that the reaction started. After about 3 hours, a polyester having a weight-average molecular weight of 65,000 was obtained.

The resulting polyester was diluted with toluene to obtain a 50% by weight solid content concentration. An adhesive composition was prepared by adding a hexamethylene diisocyanate adduct of trimethylolpropane (Coronate HL produced by Nippon Polyurethane Industry Co., Ltd.) as a crosslinking agent in an amount of 2 parts based on 100 parts of the solid content of the polyester.

A waterstop sealing material (Example 2-4) was then prepared in the same manner as in Example 2-3 in the present application, except that the adhesive composition prepared above was used instead of the polyester-based adhesive composition in Example 2-3. The sample was evaluated and the results are summarized below.

EVALUATION RESULTS

	U-shaped Watertight Test		High Pressure Bursting Watertight Test
	Immediately after mounting	After 168 hours of 80°C Aging	
Example 2-4	Water stopped at compressibility of 10%	Water stopped at compressibility of 10%	Water stopped at compressibility of 10%

EFFECTS OF THE ADHESIVE LAYER THICKNESS ON THE RESULTS

Usually, the thickness of an adhesive layer is 2 to 100  $\mu\text{m}$  (page 15, lines 19 to 20 of the specification). When the thickness is below 2  $\mu\text{m}$ , the effect of providing an adhesive layer may not be exhibited due to a decrease in adhesiveness. On the other hand, in the case of a thickness of 100  $\mu\text{m}$  or more, there is no adverse effects on the sealing property. However, sometimes productivity deterioration can occur since bubbles tend to generate in the adhesive layer and the surface tends to be roughened. Applicants wish to emphasize that within this range of thickness, the thickness of the adhesive layer never adversely affects the sealing property.

The watertight sealing material in accordance with the invention of the present application exhibits a sealing capability under compression. However, what exhibits a sealing capability under compression is the foam layer having a foamed structure; the adhesive layer does not undergo compressed deformation. As a matter of course, the adhesive strength grows between

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the adhesive material and the adhered matter by compression, thus resulting in better sealing. A sufficient adhesive strength can be attained with a pre-determined thickness.

This is clearly demonstrated by the comparison of Example 2-1 with Example 2-2. In Example 2-1, a polyester-based adhesive layer of 30 µm is provided on the outermost layer. On the other hand, in Example 2-2, a polyester-based adhesive layer of 5 µm is provided on the outermost layer. In these two examples, the watertight capability is substantially equal.

In view of the above evidence and the evidence of record, I conclude that the present invention provides unexpectedly superior results.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: 09/17/03

Katsuaki Tachibana  
Katsuaki Tachibana



PATENT APPLICATION

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In re application of

Docket No: Q57866

Katsuhiko TACHIBANA, et al.

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Group Art Unit: 1711

Confirmation No.: 2630

Examiner: Melanie D. Bissett

Filed: February 09, 2000

For: WATERSTOP SEALING MATERIAL

**DECLARATION UNDER 37 C.F.R. § 1.132**

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

I, Katsuhiko Tachibana, hereby declare and state:

THAT I am a citizen of Japan; and

THAT I am a co-inventor of the invention disclosed and claimed in the above-identified application.

I have performed or have had performed under my direct supervision and control the experimentation described below.

**EXPERIMENTATION**

Into a four-necked separable flask equipped with an agitator, a temperature indicator and a water-separating tube were charged 200 g of polycaprolactonediol which is liquid at room temperature (PLACCEL L212AL produced by Daicel Chemical Industries, Ltd.; hydroxyl number: 89.4 KOH mg/g) (hydroxyl group: 0.319 equivalent), 15.94 g of succinic anhydride

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(acid group: 0.319 equivalent), and as a catalyst 79 mg of dibutyltin oxide (abbreviated as DBTO hereinafter) (0.1 equivalent %). In the presence of a small amount of toluene as a solvent for discharging reaction water, the mixture was heated with stirring to a temperature of 180°C and then kept at the temperature. After a while, water was discharged and separated, showing that the reaction started. After about 3 hours, a polyester having a weight-average molecular weight of 65,000 was obtained.

The resulting polyester was diluted with toluene to obtain a 50% by weight solid content concentration. An adhesive composition was prepared by adding a hexamethylenediisocyanate adduct of trimethylolpropane (Coronate HL produced by Nippon Polyurethane Industry Co., Ltd.) as a crosslinking agent in an amount of 2 parts based on 100 parts of the solid content of the polyester.

A waterstop sealing material (Example 2-4) was then prepared in the same manner as in Example 2-3 in the present application, except that the adhesive composition prepared above was used instead of the polyester-based adhesive composition in Example 2-3. The sample was evaluated and the results are summarized below.

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**EVALUATION RESULTS**

	U-shaped Waterstop Test		High Pressure Running Waterstop Test
	Immediately after mounted	After 168 hours of 80°C aging	
Example 2-4	Water stopped at compressibility of 10%	Water stopped at compressibility of 10%	Water stopped at compressibility of 10%

**EFFECTS OF THE ADHESIVE LAYER THICKNESS ON THE RESULTS**

Usually, the thickness of an adhesive layer is 2 to 100 µm (page 15, lines 13 to 20 of the specification). When the thickness is below 2 µm, the effect of providing an adhesive layer may not be exhibited due to a decrease in adhesiveness. On the other hand, in the case of a thickness of 100 µm or more, there is no adverse effects on the sealing property. However, sometimes productivity deterioration can occur since bubbles tend to generate in the adhesive layer and the surface tends to be roughened. Applicants wish to emphasize that within this range of thickness, the thickness of the adhesive layer never adversely affects the sealing property.

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In view of the above evidence and the evidence of record, I conclude that the present invention provides unexpectedly superior results.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: \_\_\_\_\_

Katsuhiko Tachibana